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A Review of Advanced Reactive Materials in Fiber Sensor Technology: Challenges and Requirements of Precise Humidity Control in Mechanochemical Treatment Process

Muhammad Arif Riza, Yun Ii Go,* Ismail Zaitizila, and Soo Ping Kok

School of Engineering and Physical Sciences, Heriot-Watt University Malaysia
1, Jalan Venna P5/2, Precinct 5, 62200 Putrajaya, Malaysia

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In this comprehensive review, we explore the intersection of reactive materials in fiber sensor technology and the critical role of humidity control in mechanochemical treatment processes. We examine recent advancements in embedded sensor technologies, focusing on their applications in various treatment processes across industries. We highlight the development of metal oxide nanomaterials as effective optoelectronic humidity sensors, discussing their fabrication, characterization, and operational principles. In this review, we also delve into the emerging field of wearable chemical sensors, emphasizing their potential in healthcare monitoring and environmental applications. Furthermore, we address the integration of embedded sensors in Industry 4.0 frameworks, illustrating their impact on factory automation, process optimization, and sustainable manufacturing. We underscore the importance of humidity control in mechanochemical treatments, linking it to the broader context of sensor technology applications. By unifying current research and identifying future trends, we provide valuable insights into the synergies between reactive materials, fiber sensor technology, and humidity control, offering a foundation for further innovations in treatment process enhancement and efficiency.

1. Introduction

The world of sensor technology has undergone a remarkable evolution, driven by the ever-increasing demand for accurate and real-time monitoring across various industries. From conventional sensors to cutting-edge optical-fiber-based systems, the field has witnessed significant advancements in recent years. This progress is particularly evident in the realm of humidity sensing, where the need for precise control and measurement has become paramount in numerous applications, including mechanochemical treatment processes.(1)

*Corresponding author: e-mail: y.go@hw.ac.uk
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Mechanochemistry, a branch of chemistry that harnesses mechanical energy to induce chemical reactions and transformations, has attracted considerable attention owing to its potential for sustainable and efficient synthesis methods. This approach offers several advantages over traditional solution-based techniques, such as enabling reactions between solid reactants, eliminating the need for solvents, and simplifying scale-up procedures.

Mechanochemical treatments, which integrate mechanical approaches and chemical reactions, can significantly improve the mechanical and optical properties of nanocomposites. By fine-tuning the dispersion process, these nanocomposites have potential applications through acting as sensing materials that improve an optical fiber's ability to interact with the environment, thereby increasing the sensitivity and selectivity of the sensor.(2) The mechanochemical approach can also be used to synthesize a wide range of functional materials to alter the optical fiber's responses to physical or chemical changes for specific sensing applications, such as chemical, biological, or physical sensing.(3)

Despite the numerous advantages of the mechanochemical approach, the complexity of mechanochemical reactions and the need for a deeper understanding of mechanical forces at the molecular level have highlighted the importance of advanced sensing technologies. In particular, the control and monitoring of humidity during mechanochemical treatments have emerged as critical factors in optimizing reaction outcomes and ensuring reproducibility.(4)

The development of optical fiber sensors has revolutionized the field of sensing technology, offering numerous advantages over conventional electronic sensors. These innovative devices leverage the unique properties of optical fibers to provide fast, accurate, and highly sensitive measurements across a wide range of applications. In the context of humidity sensing, optical-fiber-based systems have demonstrated remarkable potential, with ongoing research focused on enhancing their sensitivity, resolution, and overall performance. From well-established structures such as long-period fiber gratings and fiber Bragg gratings (FBGs) to novel configurations such as resonators and sensors based on lossy mode resonances, the field continues to evolve rapidly. Furthermore, recent advancements in polymer optical fibers suggest that the full potential of these sensors has yet to be realized, promising even greater improvements in sensitivity and resolution for future humidity monitoring applications.(5,6)

2. Classification of Fiber Sensors

Fiber sensors have become a powerful and versatile tool in sensing and measurements. These sensors utilize the properties of fibers to detect and measure various physical, chemical, and biological factors. The categorization of fiber sensors is important for understanding their principles, applications, and limitations. Fiber sensors can be categorized in various ways on the basis of their designs, operational principles, and applications. Fiber optic sensors are classified on the basis of criteria, namely, the sensing mechanism, measurand, fiber type, spatial distribution, and modulation technique, as shown in Table 1.

An optical fiber sensor can be classified on the basis of its sensing mechanism, including intensity, phase, polarization, or wavelength modulation (WM), where intensity-modulation sensors changes in intensity caused by absorption, scattering, or bending losses due to external

Table 1
Fiber optic sensor classification into five categories.

Category	Type
Operating mechanism	Intensity-based sensors Phase-based sensors Polarization-based sensors Wavelength-based sensors
Measurand Type	Physical sensors Chemical sensors Biological sensors
Fiber Type	Single-mode fiber sensors Multimode fiber sensors Specialty fiber sensors (photonic crystal fibers, microstructured fibers)
Sensing location	Point sensors Distributed sensors Quasi-distributed sensors
Modulation technique	Amplitude modulation Phase modulation Wavelength modulation Polarization modulation

factors can be detected and measured.(7) These types of sensor are commonly used in tasks such as liquid level detection, pressure sensing, and structural health monitoring owing to their simplicity and cost effectiveness. Phase-modulation sensing involves detecting variations in the phase of a wave passing through a fiber. External elements such as stress, magnetic fields, or temperature can lead to birefringence in the fiber, which impacts the polarization state of light. Phase modulation (PM) is used to process signals in interferometric sensing such as the Mach–Zehnder, Michelson, and Sagnac interferometers, where the interferometric sensing techniques rely on the interference of light to measure various physical and chemical parameters.(8–11) These phase-modulation sensors find applications in several highly sensitive fields, including earthquake and structural health monitoring, in addition to distributed temperature and strain sensing.(12) Polarization-modulatioin sensors rely on changes in the polarization of light as it travels down the fiber. Owing to the effects of different external factors such as stress, magnetic fields, or temperature changes, birefringence will be induced in the fiber that will further change the polarized state of light.(13) These sensors find their applications in areas that include high sensitivity for stress or magnetic field changes, such as smart grids and magnetic field sensing. Wavelength-based sensors measure shifts in the wavelength of a light signal. The most popular example is FBG, where the periodic variation in refractive index along the fiber reflects specific wavelengths of light.(14,15) Any change in strain or temperature will shift the reflected wavelength owing to variations caused within the grating period.

Fiber optic sensors are classified into three on the basis of the measurand type: physical, chemical, and biochemical sensors. Physical fiber optic sensors are specifically used in measuring various physical parameters, including temperature, strain, pressure, displacement, and vibration. The sensors utilize the changes in the optical properties of the fiber or the attached components because of changes in the physical parameter being measured. Chemical fiber optic sensors are specifically used in the measurement and detection of chemical species or chemical measurable parameters such as pH and concentrations of gas and specific molecular compounds. Cable sensors work by measuring changes in the optical properties (e.g., absorbance, fluorescence, and refractive index) that occur when the sensor interacts with the target chemical species. Fiber optic sensors are engineered to sense biological reactions involving biological substances or processes. Normally, these sensors are a combination of biological recognition elements with an optical transduction mechanism for the detection of biomolecules, cellular processes, or physiological parameters.

Fiber optic sensors can be classified according to the optical fiber used, such as single-mode fibers (SMFs), multimode fibers (MMFs), and specialty fibers, which have distinctive qualities that make them more fitting for certain uses. SMFs have a very small core diameter compared with the wavelength (5–10 μm) and supports only one mode of light propagation along the core.[16,17] The absorption coefficients of these analytes are very large, and thus, the SMFs can be particularly well-suited for a variety of sensing applications with low-loss, large-bandwidth liquid core fibers that have been developed. SMFs are commonly used in structural health monitoring, telecommunications, and biomedical applications where high accuracy and stability are necessary. MMFs have a larger core diameter that allows multiple modes of light propagation and are more suitable for applications where robustness and cost effectiveness are less critical than precision. Specialty fibers, on the other hand, are designed with unique structures or compositions that enhance specific properties for sensing purposes. Some common types include photonic crystal fibers, polarization-maintaining fibers, and doped fibers. These types of fiber can be sensitively tailored to temperature, strain, pressure, and chemical composition sensing.[18–20]

Optical fibers have been extensively used in sensing designs because they can transmit light over long distances with low loss and because of their tolerance to environmental variation.[21,22] These applications can be divided into three main classes: point, distributed, and quasi-distributed sensing. Point sensors record parameters at a certain point along a fiber as discrete entities. The sensor element is typically a discrete device such as an FBG or a Fabry–Pérot interferometer that has been embedded within the fiber itself.[23] On the other hand, distributed fiber optic sensors enable measurements over the entire length of a fiber, thus providing a complete spatial profile. Rayleigh, Brillouin, or Raman scattering may be employed to achieve this.[24] Quasi-distributed sensors combine the sensing principles of both point and distributed sensing,[24] typically using multiple discrete sensors, such as FBGs, placed along the length of an optical fiber to allow simultaneous measurements of various parameters at multiple locations along a single fiber.

Modulation techniques in optical fibers change the properties of the light wave to encode data. The primary modulation strategies include amplitude modulation (AM), PM, WM, and polarization modulation.[5,25] AM refers to the modulation of the amplitude of an optical carrier signal, where changes in the measured parameter cause a variation in the intensity of light transmitted through the fiber. PM refers to the modulation of the phase of an optical carrier signal, where the external perturbations cause changes in the phase of the light resulting from changes in optical path length.[26] WM, also known as frequency modulation, changes the wavelength or frequency of an optical carrier signal, where the wavelength of light reflected or transmitted by a sensor varies with a measured parameter.[27] The principle of polarization modulation is that the external perturbation causes changes in the polarization state of light in optical fiber,[28] in which the orientation of the wave's oscillations changes, while the amplitude, frequency, and phase of the optical carrier signal remain the same.[29]

3. Need for Sensing Materials in Fiber Sensors

The requirement for sensing materials is an important aspect for the design and operation of fiber optic sensors. Fiber sensors have been extensively studied owing to their small size, lightweight nature, immunity against electromagnetic interference, and ability to perform distributed sensing. However, the sensing materials chosen determine how well a sensor will be performing its intended function and other factors of the sensor such as its sensitivity and application areas. The fiber sensor's sensing material is very important as it helps in converting physical or chemical changes into an optical signal that can be measured and analyzed. Most of the time, this material gets integrated with or coated on the optical fiber. The sensing material alters the properties of light passing through the optical fiber, as it interacts with the external environment. These modifications can appear as such phenomena, such as those related to light intensity, phase, polarization, wavelength, transmission, and reflection properties, which occur according to various combinations of the type of transducer and materials employed.

Fiber sensors are equipped with a range of sensing materials that enable them to detect and measure different parameters including physical, chemical, and biological ones. These materials interact with target analytes or environmental conditions leading to changes in fiber optic properties that are subsequently quantifiable and related to a specific parameter. Fiber sensors usually employ several types of sensing material, such as biomolecules,[30] polymers,[31] nanoparticles,[32] metal oxides,[19,33] plasmonic materials, and quantum dots.[34]

Specialized sensing materials are incorporated into fiber optic sensors that interact with target parameters allowing for transduction into an optical signal change. Fiber optic sensors have different applications across various areas such as gas detection, temperature control, biosensing, and chemical analysis. These types of transducer exploit a range of physical and chemical laws to convert environmental alterations into optical signals, allowing for accurate and reliable measurements in diverse sectors. It was found that a fiber optic biosensor can be used for the detection of cardiac troponin using gold nanoparticles functionalized with specific antibodies.[35] Compared with other available methods with poor selectivity, this sensor exhibited remarkable sensitivity. The sensor had good reversibility and high sensitivity for detecting ammonia within aqueous solutions. Among the many applications to which fiber optic sensors are put is temperature monitoring, which has been in use for quite some time now. Notably, these sensors may operate in various extreme situations such as high temperatures experienced in industries such as aerospace as well as power production and even others too numerous to enumerate here. This also allows accurate measurements at long distances through techniques such as the use of FBGs or Raman scattering commonly used in temperature measurement.[36,37]

Integrating judiciously chosen sensing materials with optical fibers can provide fiber sensors high sensitivity and low detection limits, selectivity for targeted analytes, versatility in measuring parameters, and miniaturization. According to a study,[38] the inclusion of carefully selected sensing materials can substantially decrease detection limits and increase sensitivity in fiber-optic sensors, allowing for measurements at lower analyte concentrations or the discovery of subtle environmental changes. An increase in the selectivity via the targeted detection of certain analytes can be achieved by carefully choosing and functionalizing sensing materials, which enables the targeted detection of certain analytes while reducing interference from other substances to obtain better specificity of the sensor.[39] Other advantages provide versatility by allowing for the detection of various parameters on a single platform. The availability of a wide array of sensing materials allows for the construction of versatile sensing platforms that can measure a wide range of parameters within a single fiber-optic system.[22,40] On the miniaturization potential, the seamless integration of sensing materials into the optical fibers will allow the development of compact, portable, and even implantable sensing devices, thus generalizing their applications in space-constrained environments and *in vivo*.[41]

In conclusion, the need for sensing materials in fiber sensors is paramount for achieving high-performance, selective, and versatile sensing capabilities. Ongoing research in materials science and nanotechnology continues to expand the possibilities for fiber-optic sensing applications across various fields.

4. Classification of Mechanochemical Treatment Processes

Mechanochemistry refers to chemical reactions and transformations induced by mechanical energy, typically through grinding, milling, or mechanical deformation. Such mechanochemical treatment processes provide an alternative route for chemical synthesis that offers several advantages compared with traditional solution-based methods. Mechanochemistry can enable reactions between solid reactants that may not be soluble, eliminate the need for solvents, reduce energy input by enabling reactions at or near ambient temperatures, simplify workup procedures, and allow easy scale-up.

A mechanical treatment activates solid reagents by fracturing their crystal lattice and generating fresh surfaces, defects, crystal strain, and thorough mixing. These effects lower reaction barriers and activate the solids for subsequent chemical reactions. Parameters such as milling intensity, frequency, time, atmosphere, and temperature can be tuned to control the mechanochemical reactions. High-energy ball milling is commonly used for mechanochemical synthesis, where ball bearings pulverize and mix the reactants. Monitoring pressure changes during milling provides insights into the reaction mechanism.[42]

Mechanochemistry has been utilized for various applications including the production of alloys, metal hydrides, nitrides, and nanomaterials such as graphene and boron nitride sheets, and even some organic reactions. With increasing focus on sustainable processes, mechanochemistry presents opportunities for solvent-free green synthesis. However, the reactions can be complex, and a greater understanding of the effects of mechanical forces at the molecular scale is still needed. Overall, mechanochemistry offers a versatile approach to inducing chemical transformations through mechanical treatments alone.[43]

5. Humidity Control in Mechanochemical Treatment Processes

Humidity control is crucial in mechanochemical treatment processes owing to its significant impact on reaction outcomes and product composition. Research indicates that water vapor can inhibit mechanochemical synthesis, as demonstrated in the glycine–malonic acid system, where varying humidity levels affect the reaction dynamics in different mechanical devices. Furthermore, humidity affects the product composition in reactions such as those between NaF and AlF_3, where increased water content promotes the formation of specific phases, highlighting the necessity of controlled humidity levels for desired outcomes. Effective humidity management can also prevent unwanted condensation in chemical reactors, which can lead to clogging and hinder gas transport. Various methods for humidity control, including the use of drying agents and salt hydrates, can stabilize reaction conditions and enhance process efficiency.(43) Thus, maintaining optimal humidity is essential for maximizing the effectiveness and reliability of mechanochemical processes.

Neglecting humidity control in mechanochemical treatment processes can lead to significant adverse effects on the synthesis process. Research indicates that the presence of water vapor inhibits mechanochemical reactions, particularly in systems like glycine–malonic acid, where varying humidity levels can alter the reaction dynamics depending on the type of mechanical device used.(44) This highlights the critical need for humidity management to ensure consistent reaction outcomes. Moreover, uncontrolled humidity levels can introduce variability in chemical processes, potentially leading to undesired interactions between reactants and products, which may compromise the efficiency and yield of the mechanochemical treatment. The thermodynamic principles governing humidity control suggest that maintaining a stable environment is essential for optimal reaction conditions. Failure to manage humidity can result in not only reduced reaction rates but also the formation of unwanted byproducts, ultimately affecting the quality of the final product. Thus, effective humidity control is vital for successful mechanochemical synthesis.(45)

6. Challenges, Requirements, and Industry Standard

Controlling relative humidity (RH) in treatment processes presents several challenges, requirements, and industry standards. One significant challenge is the fluctuation of RHs, which can adversely affect product quality and microbial survival, particularly in food processing environments where pathogens such as *Listeria monocytogenes* thrives under specific humidity conditions. Effective control systems must integrate advanced technologies, such as fuzzy logic and PID algorithms, to enhance precision and stability in RH management.(1) Industry standards necessitate the use of specialized apparatuses, such as nebulizers and humidity sensors, to maintain desired RHs in various applications, including semiconductor processing.(46) Furthermore, the requirement for real-time monitoring and control is critical, as demonstrated by systems that adjust water vapor levels in response to process demands. Overall, achieving optimal RH control is essential for improving production quality, ensuring hygiene, and enhancing energy efficiency in industrial processes.(47,48) Figure 1 shows the logo of the ISO involving RH standards in industries and Table 2 shows the descriptions of the standards.

Fig. 1. (Color online) ISO standard.

Table 2
Acceptable RH standards in industries.

ISO	RH standard description
ISO 14644-1:2015	This standard recognizes the importance of RH in preventing static electricity discharge, which can damage sensitive electronic components, and in controlling particle generation and deposition.[49]
ISO 14644-1 Class 7	This ISO states that RH should be maintained at 55%–65% for general-purpose cleanrooms.[50]
ISO 22000:2018	This highlights the importance of monitoring and controlling RH to ensure food safety and quality. Appropriate RHs are crucial for preventing microbial growth, maintaining product texture and flavor, and extending shelf life.[51]

7. Conventional Approach and Enabling Technology

RH sensing has long relied on conventional capacitive or resistive sensors, but these traditional methods often face limitations such as temperature sensitivity and hysteresis. In recent years, fiber optic sensors have emerged as an innovative and promising alternative, offering unique advantages that address many of the shortcomings of conventional approaches. These optical-fiber-based sensors leverage the intrinsic properties of light transmission to achieve enhanced performance in RH measurements. One of the most significant advantages of fiber optic sensors is their electromagnetic passivity, rendering them immune to electromagnetic interference. This characteristic makes them particularly well-suited for deployment in high-energy environments, where conventional sensors might struggle to maintain accuracy.[34] Furthermore, fiber optic sensors demonstrate remarkable sensitivity to humidity changes. For instance, FBG sensors have shown significant wavelength shifts in response to variations in RH, with smaller fibers achieving sensitivities as high as 2.7 pm/%RH.[52] This level of precision allows for highly accurate RH measurements across a wide range of environmental conditions. Another key advantage of fiber optic sensors is their capability for distributed sensing. Advanced techniques such as optical frequency domain reflectometry enable the simultaneous measurement of both RH and temperature with exceptional spatial resolution, achieving sensitivities of 1.8 pm/%RH.[24] This multiparameter sensing capability, combined with high spatial resolution, opens new possibilities for comprehensive environmental monitoring in various industrial and scientific applications.

The development of fiber optic RH sensors has been further enhanced by innovative fabrication techniques that improve both their performance and durability. One such technique is through-coating fabrication, where FBG sensors can be inscribed directly through protective coatings. This approach not only enhances sensor durability but also improves its performance without requiring extensive additional processing.[(52)] Another promising development is the creation of all-polymer microcavities, which offer rapid response times and significant miniaturization potential. These compact designs are suitable for a wide range of applications, from industrial process control to environmental monitoring.[(53)] Despite these advantages, it is important to note that fiber optic sensors still face challenges in terms of cost and complexity when compared with traditional RH sensing methods. The specialized equipment and expertise required for their production and implementation can be a barrier to widespread adoption. However, the unique benefits they offer, particularly in harsh environments where conventional sensors may fail, make them an increasingly attractive option for future RH sensing applications. As research in this field continues to advance, it is likely that we will see further improvements in both the performance and cost-effectiveness of fiber optic RH sensors, potentially leading to their broader adoption across various industries and scientific disciplines. The ongoing development of these sensors represents a significant step forward in our ability to accurately measure and monitor RH in even the most challenging environments, paving the way for more precise and reliable environmental control and research capabilities.

8. Water–Solid Interactions

8.1 Adsorption

Interactions between water and solids are crucial in various natural and industrial processes. Water–solid interactions are important in various industries for several reasons. In the pharmaceutical industry, the interactions of moisture with solid dosage formulations are crucial for understanding water-based processes, manufacturing, and predicting the solid-state stability and shelf life of pharmaceutical products. In the field of materials science, the interactions between solids and liquids play a key role in substrate wettability and spreading dynamics of liquid droplets.[(54)] Additionally, in the manufacturing, storage, and development of pharmaceuticals, water affects a wide variety of physicochemical properties of small-molecule drug substances, emphasizing the importance of water–solid interactions in this industry. Furthermore, in the field of energy–water systems, research on water–solid interfaces have a significant impact on generating a detailed understanding of adsorption for such systems. In the food industry, understanding the factors by which water associates with solids is important when the solids are exposed to water vapor.[(51)] These examples highlight the diverse significance of water–solid interactions in various industrial applications.

There are five major mechanisms of water–solid interactions, which can be separated into three groups: internalized water (crystal hydrate formation and absorption into the bulk of amorphous solids), condensed water (capillary condensation and deliquescence), and surface (adsorption) interactions. Surface interactions involve the adsorption process wherein water molecules or particles act as adsorbates that adhere to the surface of a solid or liquid material, known as an adsorbent.[55] Water molecules accumulate on the adsorbent surface via noncovalent intermolecular interactions, which generate attractive forces between the adsorbate and the surface of the adsorbent. The average number of water molecule layers covering the surface area is a commonly used to report the amount of water adsorbed on a surface. Temperature, surface area, vapor pressure, and the binding energies between solids and water all affect adsorption.[56]

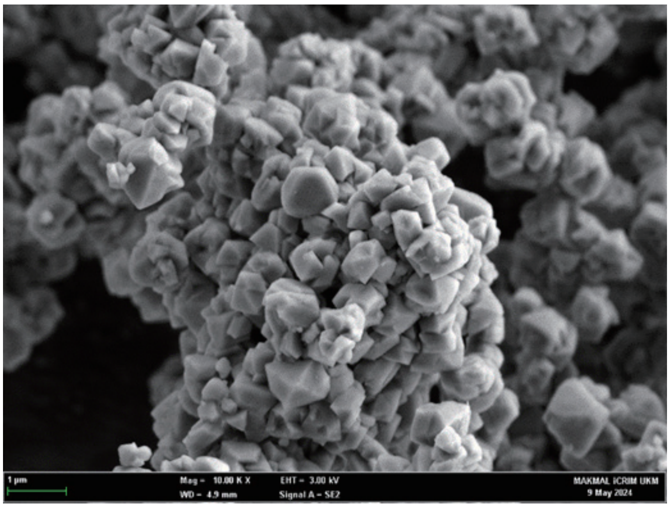
Adsorption can occur naturally via physical adsorption (or physisorption) or chemical adsorption (or chemisorption). Physical adsorption occurs owing to the van der Waals interaction between the molecules of an adsorbate and the solid adsorbent because of a weak electrostatic interaction, which is similar to the condensation of gases into liquids.[33,59] It is a reversible process that occurs at high pressures or low temperatures, and any gas tends to adsorb on any solid.[60] Examples are H_2 stripping and O_2 adsorption on charcoal surfaces.

Chemical adsorption involves stronger forces and happens when gases are bound to a solid surface by unique chemical forces (usually covalent) for every gas and surface.[60] This process is usually irreversible and occurs at higher temperatures. It is frequently a slower process and takes place at temperatures higher than those in physical adsorption. Adsorption is affected by several factors, including the nature of the adsorbent and the adsorbate, temperature, pressure, the surface area of the adsorbent, and the presence of other substances in the environment.

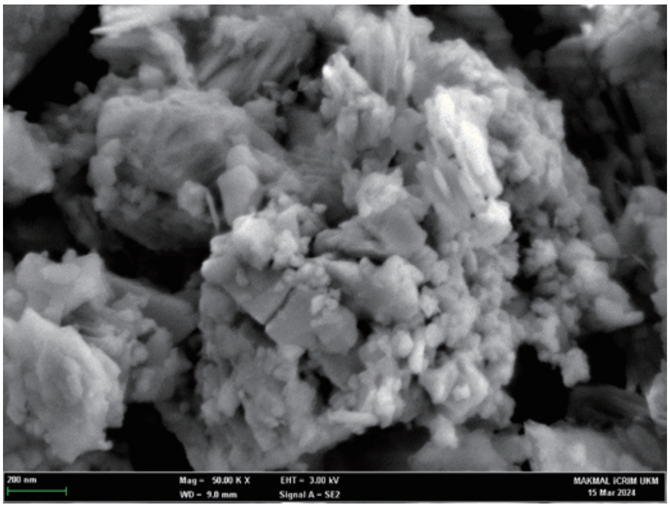
The nature of the adsorbent refers to its surface properties, such as porosity, surface chemistry, and charge, whereas the nature of the adsorbate, including its size, polarity, and concentration, also plays a significant role in adsorption.[60] Temperature and pressure affect adsorption by influencing the kinetic energy of the molecules and the equilibrium between adsorbed and unabsorbed molecules. Generally, higher temperatures and lower pressures promote adsorption. The surface area of the adsorbent is crucial, as it provides more sites for adsorption to occur. Finally, other substances present in the environment can compete for adsorption sites or interact with the adsorbent and adsorbate, affecting the overall adsorption process.[36] Figures 2(a) and 2(b) show an example of hygroscopic zeolite where the rough surface area serves as sites for water molecule attachment.

An adsorption isotherm is a fundamental tool for understanding the adsorption process and is used to determine the adsorption capacity, surface area, and pore size distribution of an adsorbent.[61] It describes the relationship between the amount of adsorbate adsorbed on the surface and the equilibrium concentration of the adsorbate in the bulk phase. There are several types of adsorption isotherm, including the Langmuir, Freundlich, and BET isotherms, which are commonly used in various fields, including chemistry, materials science, and environmental science.[62] The Langmuir isotherm is a semi-empirical isotherm that is applicable to gases adsorbed on solid surfaces, whereas the Freundlich isotherm is used to describe the adsorption of gases on heterogeneous surfaces. The BET isotherm is used to describe the adsorption of gases on porous materials. Understanding adsorption isotherms is essential in various fields, including the design of adsorption processes, the development of adsorbents, and the optimization of adsorption systems.[63]

Deliquescence is a first-order phase transition process by which a solid absorbs water from the air to dissolve itself and form an aqueous solution that occurs at an RH that depends on the properties of the solid and the temperature.[56] This process occurs when the vapor pressure of



(a)



(b)

Fig. 2. Morphology of a hygroscopic zeolite formed through adsorption-based interactions: (a) synthetic zeolite and (b) natural zeolite. (Author's Own Work)[57,58]

the substance's solution becomes equal to or greater than the partial pressure of water vapor in the surrounding air. When a crystalline substance reaches the threshold vapor pressure for ambient RH, it is said to deliquesce and form a solution. The deliquescence RH (RH0) (also known as the critical RH) is a key property that determines the ability of crystalline solids to take up water. RH0 refers to the minimum RH at which a crystalline solid absorbs enough water from the surrounding air to form a saturated aqueous solution and become thermodynamically stable as the crystalline solid.[64,65] Deliquescence is commonly observed in hygroscopic substances, which have a strong affinity for water. Examples of deliquescent substances include salts, such as sodium hydroxide (NaOH), potassium hydroxide (KOH), magnesium chloride ($MgCl_2$), ammonium chloride, gold (III) chloride, sodium nitrate, and calcium chloride ($CaCl_2$).

Deliquescence is a significant water–solid interaction for crystalline powders, which modifies their chemical and physical reactivities. Water interacts with a solid through the mechanism of adsorption when the ambient RH is lower than RH0. Both the type of crystalline compound and the environmental temperature affect RH0.[66] Highly crystalline solids usually only adsorb a small number of water molecules. For instance, sodium chloride adsorbs two to three monolayers of water below RH0, and no bulk dissolution is observed.[67] More vapor is adsorbed at the surface as the RH rises, and eventually, a thin layer of the solid's saturated solution forms on the particle when the surrounding RH surpasses the solid's RH0. The deliquescence process is based on the production of a coating of saturated solution with a lower vapor pressure than pure water over the surfaces of water-soluble particles.[56]

In summary, deliquescence is the process by which a substance absorbs water from the air to dissolve itself and form an aqueous solution. Deliquescent materials are hygroscopic substances that can absorb a large amount of water and are sufficiently soluble to dissolve in it. Deliquescence is a first-order phase transformation of a crystalline solid to a solution above a critical RH, and deliquescent materials are used as desiccants in various industries.

8.2 Capillary condensation

Capillary condensation refers to the phenomenon where a gas or vapor can spontaneously condense and form a liquid in narrow spaces, such as the pores or channels of a solid material.[68] The unique aspect of capillary condensation is that vapor condensation occurs below the saturated vapor pressure of the pure liquid.[56] This phenomenon is driven by the attractive forces between gas molecules and solid surfaces, which can include the van der Waals force, hydrogen bonding, or electrostatic interactions. These interactions between gas molecules and solid surfaces decreases the vapor pressure inside the narrow spaces, leading to an accumulation of the liquid phase in those spaces. The amount of water that accumulates because of capillary condensation is considerably dependent on the RH, type of solid, radius of capillaries, quantity of capillaries, packing density of a powder bed, particle size, surface area, and temperature. Capillary condensation is a significant element in both naturally occurring and manmade porous structures. This process is of considerable importance in various fields, including materials science, nanotechnology, and environmental engineering.

8.3 Internalized water

8.3.1 Absorption

The similar spelling of absorption and adsorption may cause some misunderstanding between these two waters–solid interactions; however, they are easily distinguished by the site of the contact. Absorption is the process by which one substance enters the internal structure of another substance.[33,69] It can occur in a variety of settings spanning various disciplines, including chemistry, physics, biology, and engineering. According to IUPAC 2006, absorption is the process by which water vapor permeates an amorphous substance. This absorption occurs owing to the porous nature of certain solids or their ability to form hydrogen bonds with water molecules. In materials science, absorption typically refers to the process by which a substance, such as a solid or liquid, takes in another substance through permeation. In the context of water absorption in polymers or amorphous solids, this can involve water molecules entering the material's matrix, potentially leading to swelling, a change in physical properties, or hydrolytic degradation. For crystalline materials such as crystal hydrates, the term absorption can be considered when water molecules become part of the crystal structure itself.

8.3.2 Crystal hydrate

A crystal hydrate, also known simply as a hydrate, is a type of chemical compound that contains water molecules within its crystal structure in which the water molecules are chemically bound to the atoms or ions of another substance at a definite stoichiometric ratio. Monohydrates (1 water molecule: 1 compound molecule) to decahydrates (10 water molecules: 1 compound molecule) provide for a variety of stoichiometric ratios of compound to water molecules. Water molecules are not chemically bonded to compound molecules but are held within the crystal lattice by weak intermolecular forces.(70) In a crystal hydrates, water molecules are incorporated into the crystal lattice of the compound, forming a distinct crystalline structure. A crystal hydrate is a solid crystalline chemical that contains water molecules as an inherent part of the crystal lattice structure, whereas its anhydrous version contains no water.(68) The amount of water molecules in a crystal hydrate can vary, and the water content is often expressed as a ratio of water to the compound.

Crystal hydrates can be found in both inorganic and organic compounds and can be classified on the basis of the number of water molecules present in the crystal structure. Crystalline hydrates are classified into three types, differing in the location of water relevant to the crystal lattice: isolated site hydrates, channel hydrates, and ion-associated hydrates. These hydrates can be classed as either stoichiometric or nonstoichiometric. Stoichiometric hydrates contain a fixed molar amount of water, whereas nonstoichiometric hydrates are channel hydrates that can incorporate additional water molecules without changing to another polymorph.(70) Many crystal hydrates can exchange water with the atmosphere under well-defined temperature, pressure, and RH. The study of crystal hydrates is important in various fields, including materials science, chemistry, and pharmaceuticals.

9. Classification of Hygroscopic Materials

Hygroscopic materials literally mean water-seeking substances. These are materials that show a very high affinity for water molecules that exist in the surroundings, freely absorbing moisture from air. This absorption can happen through direct contact with liquid water or by attracting water vapor from the atmosphere. Examples of hygroscopic materials include wood, paper, silica gel, honey, methanol, concentrated sulfuric acid, glycerin, ethanol, and various salts such as sodium chloride, CaF_2, ferric chloride, $MgCl_2$, zinc chloride, potassium carbonate (K_2CO_3), KOH, and NaOH.(71) Hygroscopic materials are used in various applications such as drying agents for moisture-sensitive products, adsorbents, humectants, and dehumidifiers. The qualification of hygroscopic and nonhygroscopic materials is determined on a case-by-case basis, with materials that pick up more than 5% by mass between 40 and 90% RH at 25 ℃ described as hygroscopic, whereas materials that pick up less than 1% under the same conditions are regarded as nonhygroscopic.(72) In this paper, hygroscopic materials refer to porous metal oxides, polymers, and organic and inorganic materials.

Zinc oxide can be referred to as a material with diverse applications owing to its unique physical and chemical characteristics. Zinc oxide is a hygroscopic material with quite a good number of uses such as humidity sensors, rubbers, ceramics, glass, cement, lubricants, paints, adhesives, and sunscreens, among many other uses.(68,73) Its unique hygroscopic properties make zinc oxide an ideal material for humidity sensors owing to its ability to pick up moisture from the environment and change its electrical conductivity in the process. This property allows the measurement of humidity levels in different areas, from industrial to agricultural and residential. Moisture sensitivity makes it possible to achieve high-precision and -reliability humidity sensing. Scheme 1 is one of the synthesis routes for porous hygroscopic ZnO.

The hygroscopic nature of copper oxide (CuO) allows it to easily and effectively absorb and regulate moisture in a wide range of settings. This substance is used in the production of desiccants for products that are sensitive to moisture, which include pharmaceuticals and food. Its application in industrial process humidity control systems enables the maintenance of optimum levels of moisture, hence ensuring better quality and increased lifespans of products. The hygroscopic properties of copper oxide have been a proof for its potential applications in the reduction of moisture-based degradation across various industries. Other than being substantially effective in controlling humidity, its effectiveness has already been proved in many practical fields, making it a reliable solution for managing moisture. The importance of copper oxide as a hygroscopic material is its unique properties and its wide-ranging applications in moisture control across various industries, making it, so far, an important element in the quest for the regulation of humidity levels. Of these, the hygroscopic properties of copper oxide prevail as the most promising materials in humidity sensing applications.(74)

Aluminum oxide, or alumina, with the chemical formula Al_2O_3, is hygroscopic in nature and thus can absorb and retain some moisture from the atmosphere. This is due to hydroxyl groups on the surface of aluminum oxide binding with water molecules to form surface hydroxides. This property has implications in real-world applications, particularly in industries where moisture control is vital, such as pharmaceuticals and food processing. The capacity of aluminum oxide to absorb moisture makes it helpful in desiccants and humidity control systems, maintaining the integrity and shelf life of delicate products. Alumina-based desiccants as shown

$$Zn(CH_3COO)_2 \rightarrow Zn^{2+} + CH_3COO_2 \text{ (Dissolution reaction)}$$
$$NaOH \rightarrow Na^{2+} + 2OH^-$$
$$Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2$$
$$Zn(OH)_2 \rightarrow ZnO + H_2O \text{ (ZnO nanoparticle formation)}$$

Scheme 1. Formation of porous hygroscopic zinc oxide.(18)

in Fig. 3 have been found to effectively control moisture levels in pharmaceutical formulations, highlighting the significance of aluminum oxide in this industry. Alumina's hygroscopic nature allows it to regulate moisture levels in food products, preventing spoilage and maintaining freshness.(69,75) Figure 4 shows the thermal gravimetric analysis (TGA) of a porous hygroscopic material losing its mass owing to the evaporation of surface water.

10. Classification of Deliquescent Materials

Deliquescent materials are substances that can absorb moisture from the environment and transition from a solid to a liquid state when the RH reaches a specific threshold, known as the RH0. These materials include various inorganic salts, sugar alcohols, and amino acids, which exhibit significant water adsorption characteristics, particularly at high humidity. The deliquescent behaviour is affected by the solubility of the compounds; higher solubility typically correlates with lower RHs. Research indicates that deliquescent materials can enhance reaction kinetics in thermochemical heat storage applications by improving ionic mobility through their

Fig. 3. (Color online) Hygroscopic powders packed on an analytical boat.

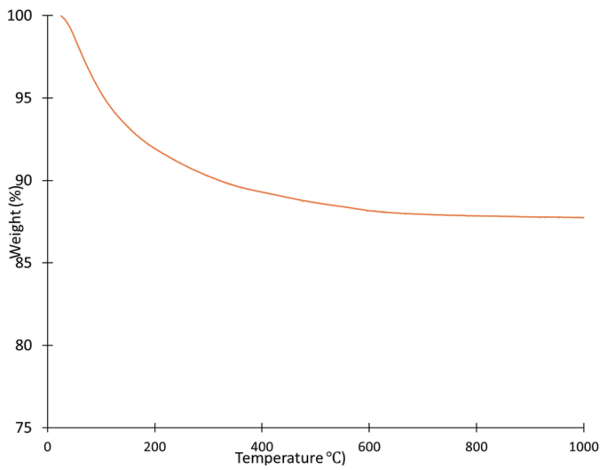


Fig. 4. (Color online) TGA response of porous silicate hygroscopic material.

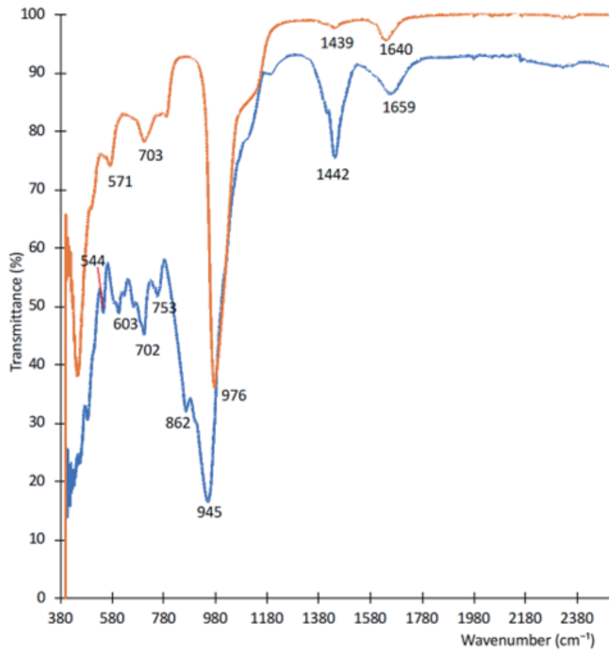
hygroscopic nature.[76] Additionally, materials such as calcium phosphate have demonstrated substantial water uptake and rapid release capabilities, showcasing their potential for water capture and purification. Furthermore, deliquescent chromism in nickel (II) iodide thin films illustrates the unique optical properties that can arise from deliquescence, expanding the functional applications of these materials. Overall, deliquescent materials play a crucial role in various fields, including food stability, energy storage, and advanced materials science.

Deliquescent materials are substances that can absorb moisture from the atmosphere and transition into a liquid state under specific humidity conditions. Examples of deliquescent materials are various inorganic salts, sugar alcohols, and amino acids, which exhibit significant water adsorption properties at certain RH thresholds, leading to structural transformations into a liquid phase. Specific salts such as K_2CO_3 and lithium sulfide (Li_2S) are also noted for their deliquescent properties, enhancing their reactivity and utility in thermochemical heat storage and battery applications, respectively.[77] Additionally, calcium phosphate materials synthesized via sol–gel techniques demonstrate deliquescent properties, showing high water uptake and maintaining structural integrity post-deliquescence. Nickel(II) iodide (NiI_2) thin films exhibit a unique deliquescent chromism, changing color in response to humidity, which highlights the diverse applications of deliquescent materials in technology.[78] These findings underscore the versatility and significance of deliquescent materials across various fields. Figure 5 shows FTIR spectra of lab-based and natural hygroscopic materials. Natural hygroscopic materials tend to have more hydrophilic O–H groups and thus can attract water molecules toward the nanostructure surface.

11. Embedded Sensor Technology for Performance Enhancement in Treatment Process

In the rapidly evolving landscape of industrial processes and technological advancements, embedded sensor technology has emerged as a crucial component for enhancing performance and efficiency in various treatment processes. This innovative approach integrates sophisticated sensing capabilities directly into the core of operational systems, allowing for real-time monitoring, analysis, and control. The application of embedded sensors spans across multiple domains, from environmental monitoring to healthcare and industrial automation, offering unprecedented levels of precision and responsiveness in process management.[79]

One of the most significant applications of embedded sensor technology in treatment processes is in the realm of humidity control and detection. As highlighted in recent studies, metal oxide nanomaterials have shown exceptional promise as optoelectronic humidity sensors. These sensors, fabricated as thin or thick films based on semiconducting metal oxides, leverage the n-type conduction properties and refractive index changes of these materials when exposed to moisture. This sensitivity allows for highly accurate humidity detection, crucial in various industrial and environmental applications. The versatility of these sensors is evident in their adaptability to different substrates and light sources, enabling their use in remote or unmanned stations where traditional monitoring methods may be impractical or impossible.[79]



Wavenumber (cm-1)	Assignment
440-450	bending vibrations – δ O-Si-O, occurring in "antiphase"
595-610	symmetrical stretching vibrations Si–O–(Si,Al)
794-810	asymmetric and symmetrical stretching vibrations corresponding to the external TO4 structure (T = Si, Al)
1011-1025	asymmetric stretching vibrations Si–O–Si and Si–O–Al
1627-1640	OH stretching vibrations
3395-3043	bridged Si– O(H)–Al and the silanol Si–OH groups perturbed by a strong H-bonding

Fig. 5. (Color online) FTIR spectra of hygroscopic materials. The blue curve is for a natural aluminosilicate hygroscopic material, and the orange curve is for a synthetic lab-grown aluminosilicate.

The integration of embedded sensors into wearable technologies represents another frontier in treatment process enhancement, particularly in healthcare and environmental monitoring. Recent innovations have led to the development of wearable chemical sensors capable of detecting various markers in bodily fluids such as sweat, tears, and saliva. These sensors, often designed as patches, tattoos, or microfluidic systems, offer non-invasive methods for continuous health monitoring and early disease detection. The evolution of these technologies is moving toward multifunctional, sensing–therapeutic systems that not only monitor but also respond to detected conditions, exemplified by advancements in microneedle technology for drug delivery. This trend toward integrated, self-powered systems marks a significant step in creating closed-loop sensing and treatment processes, potentially revolutionizing personalized healthcare and environmental safety monitoring.[80]

In the context of industrial applications, particularly within the frameworks of Industry 4.0, embedded sensors are playing a pivotal role in advancing factory automation and process optimization. These sensors, ranging from position and pressure sensors to temperature and force sensors, enable the real-time detection, measurement, and analysis of various physical parameters critical to manufacturing processes. The implementation of these smart sensing technologies contributes to enhanced sustainability, improved production mobility, and reduced maintenance costs in smart factories. As Industry 4.0 continues to evolve, the integration of embedded sensors with IoT and wireless sensor networks is paving the way for more sophisticated, efficient, and responsive treatment processes across various sectors, including agriculture, where smart farming systems are being developed to address the growing global food demand. This convergence of embedded sensor technology with advanced data processing and automation techniques have potentials to unlock new levels of performance and efficiency in treatment processes, driving innovation and sustainability across multiple industries.[81]

12. Conclusions

This comprehensive review has illuminated the significant interplay between reactive materials in fiber sensor technology and the crucial role of humidity control in mechanochemical treatment processes. The examination of recent advancements in embedded sensor technologies has revealed their wide-ranging applications across various industries. Metal oxide nanomaterials have emerged as highly effective optoelectronic humidity sensors, with their fabrication, characterization, and operational principles thoroughly explored. In this review, we also highlighted the promising field of wearable chemical sensors, particularly their potential in healthcare monitoring and environmental applications. The integration of embedded sensors within Industry 4.0 frameworks has demonstrated substantial impacts on factory automation, process optimization, and sustainable manufacturing. Furthermore, the importance of humidity control in mechanochemical treatments has been underscored, establishing its connection to the broader context of sensor technology applications. By synthesizing current research and identifying future trends, we have provided valuable insights into the synergies among reactive materials, fiber sensor technology, and humidity control. These findings lay a solid foundation for future innovations in treatment process enhancement and efficiency, paving the way for continued advancements in this dynamic field.

Conflicts of Interest

There are no conflicts to declare.

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